Control of Cooked Flavor in High-Temperature Short-Time Milk Concentrates with a Sulfhydryl-Blocking Agent

Pilot plant experiments confirmed laboratory findings on the ability of organic thiolsulfonates to suppress the cooked flavor in high-temperature short-time (HTST) sterile milk concentrates. Specifically, the flavor of treated samples was improved markedly with respect to that of their untreated counterparts when 2-acetamidoethyl 2-acetamidoethanethiolsulfonate (AETS), currently not on the list of approved food additives, was added at the rate of 5 mg/100 ml of reconstituted whole milk in the operational sequence: forewarming, concentration, sterilization, homogenization, addition of AETS, and canning. Comparative keeping qualities (up to 85 days) of control and treated samples were determined at 4.4 and 21° by a panel of selected judges. Using a 10-point scale, ranging from 31 to 40, the average score of freshly made treated samples was about 3 units higher than that of freshly made untreated samples. The score difference tended to decrease slowly as the storage time increased and other off-flavors (mostly "stale") arose. No additional foreign flavors were observed in the samples treated with AETS.

Flavor problems that develop during processing and storage of high-temperature short-time (HTST) and ultra-high-temperature (UHT) sterile milk concentrates are a major obstacle to their commercialization as satisfactory beverages. The distinct cooked flavor present in recently made sterile milks fades on storage, and is replaced by another less clearly defined off-flavor which is often described as "stale." Much evidence shows that the cooked flavor is due to volatile sulfur compounds, primarily thiols, that arise on thermal breakdown of serum proteins and of the proteinaceous material associated with the fat globule membrane (Hutton and Patton, 1952; Josephson, 1954; Patton, 1958). Some speculative pathways which ascribe a key role to the Strecker degradation (Schönberg and Moubacher, 1952), and which contemplate the participation of certain natural milk constituents or of intermediate products of the Maillard reaction (Ellis, 1959; Maillard, 1912), were discussed previously (Ferretti, 1973).

A search of the literature failed to reveal any serious attempt to control cooked flavor in fluid dairy products by chemical means. Ferretti (1973) showed that the use of sulfhydryl-blocking agents, such as organic thiolsulfonates and thiosulfates, can reduce significantly the intensity of the cooked flavor that developed when milk was heated at 90° for 5 min at atmospheric pressure. Next we wanted to determine whether the same chemicals would be equally effective under pilot plant conditions.

EXPERIMENTAL SECTION

Equipment, Materials, and Methods. Plant equipment consisted of the following units: a Wiegand-Harris tubular falling film evaporator; a Mallory-type sterilizer consisting of heating, holding, and cooling sections; a high-pressure homogenizer operating at 210 kg/cm^2 and at 64°. Products were canned aseptically in a presterilized glove box fitted with a 20-l. Pyrex glass reservoir for collecting the sterile concentrate, and equipped with a 50-ml stainless steel buret and a can seamer.

Milk was obtained from the Beltsville, Md., dairy herd (U. S. Department of Agriculture). The polyphosphate used to control gelation (Leviton *et al.*, 1963) was sodium hexametaphosphate (Fisher Scientific Co., Fairlawn, N. J.). The cooked flavor inhibitor was 2-acetamidoethyl 2-acetamidoethanethiolsulfonate (AETS) which had been the most effective in the preliminary study (Ferretti, 1973); it was prepared as reported previously (Field *et al.*, 1961).

Procedure. Two sequences were used for HTST processing: (1) forewarming, concentration, addition of AETS, sterilization, homogenization, and canning; this will be referred to as sequence 1; (2) forewarming, concentration, sterilization, homogenization, addition of AETS, and canning; this will be referred to as sequence 2.

In processing according to sequence 1, milk was fore-

warmed at 124° for 15 sec, cooled to about 70°, and pumped into the Wiegand evaporator. The total solids content of the collected concentrate, which generally was between 37 and 40%, was adjusted to 36% by addition of water. Then sodium polyphosphate was added at the rate of 0.64 kg/100 kg of milk solids. Finally, a calculated volume of a 2% aqueous solution of 2-acetamidoethyl 2-acetamidoethanethiolsulfonate was added corresponding to 3.8 mg/100 ml of reconstituted whole milk (12.5% total solids). The concentrate was then forced through the Mallory heat exchanger where it was sterilized at 140° for 10 sec. After cooling to 64°, the fluid was passed through the high-pressure (210 kg/cm^2) homogenizer and then further cooled to room temperature prior to introduction into the glass reservoir. From the reservoir, the sterile homogenized concentrate was transferred, *via* a buret, into several 220-ml cans which were sealed. The canned products were stored at 4.4 and 21°.

When processing followed sequence 2, forewarming, concentration, standardization to 36% total solids, and addition of polyphosphate were conducted as indicated above. Then, after sterilization (140° for 10 sec) and homogenization (210 kg/cm^2 at 64°), the sterile concentrate was pumped into the glass reservoir and finally transferred into the cans where the calculated amount of sterilized AETS was added just before seaming. The additive was dissolved either in 70% aqueous ethanol or in deionized water, and was sterilized by passage through a Nalgene filter unit (0.45-μ grid membrane, Sybron Corp., Rochester, N. Y.). One-half milliliter of a 6.4% (w/v) aqueous or hydroalcoholic solution of AETS was placed in each can with a calibrated pipet. Because the capacity of each can was 220 ml, the concentration of AETS corresponded to 5 mg/100 ml of reconstituted whole milk. The cans were finally sealed and stored at 4.4 and 21°.

In each pilot plant run, by either sequence 1 or 2, control concentrates were prepared without AETS and stored at 4.4 and 21°.

Flavor Evaluation. At the time of tasting, the control and the treated concentrates were allowed to equilibrate to room temperature, and then were diluted to 12.5% total solids and submitted in duplicate to a panel of six expert judges. With the experimental samples, fresh homogenized milk from the local supermarket was submitted in single or duplicate samples. The judges used the 0–4 rating scale to register the intensity of the cooked flavor (4 = strong, 3 = distinct, 2 = slight, 1 = questionable, and 0 = no criticism), and a modified American Dairy Science Association (ADSA) scorecard (Liming, 1966) for comprehensive flavor evaluation. In both cases scores from each taste panel session were averaged by dividing the sum total of scores for identical samples by the number of those scores. Such averages are taken as the basis for the discussion below.

RESULTS AND DISCUSSION

Addition of AETS before Sterilization (Sequence 1). Processing according to this sequence was abandoned because AETS did not inhibit cooked flavor as effectively as we had expected on the basis of previous work (Ferretti, 1973). Intensity of cooked flavor in the treated samples dropped only by 1 point in the 0–4 rating scale; possibly AETS was destroyed by heat during sterilization.

Addition of AETS after Sterilization (Sequence 2). When 2-acetamidoethyl 2-acetamidoethanethiolsulfonate, in 75% aqueous ethanol, was added to the sterile concentrate, the cooked flavor was almost entirely suppressed, but most judges detected novel foreign flavors. Such off-flavors were described as "astringent," "bitter," or "fruity." We suspected that the astringent and the bitter notes might be due to the ability of some of the judges to detect the ethyl alcohol. Actually, Keith and Powers (1968) reported the flavor threshold of alcohol as 53 ppm *in water.* The concentration of ethanol in the sterile reconstituted and AETS-treated milk was about 450 ppm, probably much above its flavor threshold *in milk.* The fruity off-flavor might have resulted from the formation of traces of ethyl esters by reaction of the added alcohol with short-chain fatty acids always present in the free state even in raw milk. All of the above foreign flavors were absent when AETS was dissolved in deionized water.

Scores indicated that control of cooked flavor was much better with sequence 2 than with sequence 1. This difference was not attributed to the slightly higher level of AETS—5 mg against 3.8 mg per 100 ml of reconstituted milk—but rather to its heat-accelerated decomposition during sterilization when sequence 1 was used (Field *et al.,* 1964).

Comparative Keeping Qualities of Treated and Untreated Concentrates. The samples of sterile concentrates, stored at 4.4 and 21°, were submitted to the panel of judges weekly, beginning 1 day after manufacture. Essentially, "cooked" was the only criticism expressed by the judges during the early part of the storage period. Originally, the 0–4 rating scale was used to express the intensity of the cooked flavor. One day after manufacture, the intensity of the off-flavor was 3.7 in the control samples and only 0.8 in the AETS-treated samples, a difference of almost 3 points. After 22 days' storage the difference dropped to 1.0 point in the samples stored at 4.4°, and to 1.4 in those stored at 21°. As the storage time increased, however, it became increasingly evident through the comments of the judges that other off-flavors developed during storage. These were described as "stale," "scorched," or "typical evaporated." Consequently, in the remaining sessions, and in the sessions conducted with milk from subsequent experiments, the ADSA scorecards were used to indicate the product's overall acceptability as a beverage. With this scoring system, and 8 days after manufacture, flavor scores of control and treated samples were 34.7 and 37.5, respectively, when stored at 4.4°, and 34.5 and 36.2, respectively, when stored at 21°. As storage time increased, however, the differences in scores tended to taper off. After 54 days' storage at 21° the control samples scored 34.0 and the treated samples scored 35.2; after 85 days' storage at 4.4° the scores of control and treated samples were 34.9 and 35.3, respectively. Other findings pertaining to the comparative keeping qualities of treated and untreated concentrates are summarized below.

(a) AETS-treated (A) samples always scored higher than controls (C), regardless of time and temperature of storage. (b) A samples stored at 4.4° invariably scored higher than their counterparts stored at 21°. Without AETS (C samples), storage temperature had little effect on the flavor quality. (c) Flavor scores of A samples remained *above 35* throughout the testing period, which was 54 days for those stored at 21°, and 85 days for those stored at 4.4°; on the other hand, the scores of C samples remained *below 35* (at 35 in one case) throughout the testing period. (According to the scoring system used, 40 is a perfect score, and 35 is generally considered the limit of acceptability as a beverage.) (d) The score differences among A samples were uniformly greater than the score differences among C samples; this probably was due to the substantially lower initial score of the C samples.

SUMMARY AND CONCLUSION

Results of the present work indicate that the AETS-treated HTST concentrate, prepared according to sequence 2 (AETS added after sterilization), makes a more desirable beverage milk than the untreated concentrate prepared by the same processing sequence. Furthermore, these same results underscore the promise of 2-acetamidoethyl 2-acetamidoethanethiolsulfonate, and conceivably of similar additives, as effective inhibitors of the cooked flavor in HTST sterile milks. Particularly encouraging

was the lack of introduction of foreign flavors along with AETS when this was handled in water solution. Although AETS is not presently included in a list of approved food additives, the results are encouraging enough to warrant experimentation on a larger scale.

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